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चाँदी-ताँबा टाँका मिश्रधातु का विश्लेषण

भाग 3 निकल का निर्धारण

Indian Standard

CHEMICAL ANALYSIS OF SILVER-COPPER BRAZING ALLOYS

PART 3 DETERMINATION OF NICKEL

ICS 77.120.01

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

April 2001

Price Group 1

Methods of Chemical/ Instrumental Analysis of Non-Ferrous Metals Sectional Committee, MTD 28

FOREWORD

This Indian Standard (Part 3) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Chemical Anaylsis of Non-Ferrous Metals Sectional Committee had been approved by the Metallurgical Engineering Division Council.

IS 2927 : 1975 'Specification for brazing alloys'covers 22 grades of silver-copper brazing alloys. In order to determine correctly the composition of different copper-silver brazing alloys, method of analysis have been described in various parts of this standard.

The determination of silver and copper in silver-copper brazing alloys except BACuAg7 and BACuAg8 is covered in IS 4667 (Part 1) : 1968 'Methods of chemical analysis of silver copper brazing alloys: Part 1 Analysis of silver and copper'.

The determination of silver, copper and tin in BACuAg 7 and BACuAg8 alloys are covered in IS 4667 (Part 2) : 1969 'Methods of chemical analysis of silver copper brazing alloys: Part 2 Determination of silver, copper and tin'.

The determination of silver, copper, cadmium and zinc by electrolytic method are covered in IS 4667 (Part 4) : 2001 'Chemical analysis of silver-copper brazing alloys: Part 4 Determination of silver, copper, cadmium and zinc — Electrolytic method'.

This part covers the determination of nickel by dimethylglyoxime method.

In reporting the result of a test or analysis made in accordance with this standard, if the final value observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

CHEMICAL ANALYSIS OF SILVER-COPPER BRAZING ALLOYS

PART 3 DETERMINATION OF NICKEL

1 SCOPE

This standard (Part 3) describes the method for determination of nickel in silver-copper brazing alloys in the range from 0.5 to 3.5 percent by the dimethylglyoxime method.

2 REFERENCES

The Indian Standards listed below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreement based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
266 : 1993	Sulphuric acid (*third revision*)
1070 : 1992	Reagent grade water — Specification (*third revision*)
2927 : 1975	Specification for brazing alloys (*first revision*)
4667 (Part 1) : 1968	Methods of chemical analysis of silver copper brazing alloys: Part 1 Analysis of silver and copper

3 SAMPLING

The sample shall be drawn and prepared as per the method specified in IS 2927.

4 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and reagent grade water shall be employed for the test (*see* IS 1070).

5 DETERMINATION OF NICKEL BY DIMETHYLGLYOXIME METHOD

5.1 Outline of the Method

After removal of silver, copper and cadmium, nickel is precipitated by dimethylglyoxime, filtered, dried and weighed.

5.2 Reagents

5.2.1 *Sulphuric Acid* (*see* IS 266).

5.2.2 *Hydrogen Sulphide* — gas.

5.2.3 *Hydrogen Sulphide Wash Solution* — Acidify 1 000 ml of distilled water with 10 ml of sulphuric acid and saturate with hydrogen sulphide gas.

5.2.4 *Dilute Hydrochloric Acid* — 1 : 1 (*v*/*v*).

5.2.5 *Tartaric Acid Solution* (250 g/l) — Dissolve 250 g of tartaric acid in 600 ml of water and dilute to 1 litre with water.

5.2.6 *Ammonium Hydroxide Solution* — rd = 0.90.

5.2.7 *Dimethylglyoxime Solution* (10 g/l) — Dissolve 10 g of dimethylglyoxime in ethanol and dilute to 1 litre with ethanol.

5.3 Procedure

5.3.1 To the electrolyte after removal of silver and copper as per **5.4.3** of IS 4667 (Part 1) : 1968, add 8 ml of sulphuric acid and evaporate to dense white fumes. Cool to room temperature, dilute to about 100 ml with water and heat to boiling and pass a rapid stream of hydrogen sulphide gas for about 20 to 30 min. Allow the precipitate to settle for about 3 to 4 h.

5.3.2 Filter through medium textured filter paper and wash with hydrogen sulphide wash solution for 5-6 times. Preserve the filtrate.

5.3.3 Dissolve the precipitate in warm dilute hydrochloric acid (1 : 1) and collect the solution in the original beaker in which cadmium was precipitated. Wash the paper thoroughly with hot water till acid free. Add 8 ml of sulphuric acid and evaporate to white fumes, cool to room temperature and dilute to about 100 ml. Heat the solution to boiling. Reprecipitate the cadmium by passing a rapid stream of hydrogen sulphide gas for about 20-30 min. Allow the precipitate to settle.

NOTE — Proper safety precaution shall be taken while handling hydrogen sulphide gas.

5.3.4 Filter through medium textured filter paper and wash hydrogen sulphide wash solution for 7-8 times. Preserve the filtrate.

5.3.5 Combine the filtrate from **5.3.2** and **5.3.4** and boil to remove hydrogen sulphide. Adjust the volume to about 150 ml and add 10 ml of tartaric acid solution, neutralize with ammonium hydroxide solution, add 10 ml of ammonium hydroxide solution in excess and heat to about 80°C.

5.3.6 Add 25 ml of dimethylglyoxime solution, stir vigorously and allow to cool to room temperature, stirring occasionally. Let the precipitate settle overnight.

5.3.7 Filter the precipitate through a 30-ml sintered glass crucible of medium porosity and wash with cold water. Preserve the filtrate for the estimation of zinc.

5.3.8 Dry the precipitate at 150°C to constant mass. Cool in a desiccator and weigh as nickel dimethylglyoxime.

5.4 Calculation

$$\text{Nickel, percent by mass} = \frac{m \times 0.203\,2}{M} \times 100$$

where

m = mass, in g, of nickel dimethylglyoxime; and

M = mass, in g, of sample taken.

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This Indian Standard has been developed from Doc : No. MTD 28 (3887)

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Printed at New India Printing Press, Khurja, India